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# DESIGN AND DEVELOPMENT OF REGENERATIVE CARBON DIOXIDE SORBERS

TECHNICAL DOCUMENTARY REPORT NO. AMRL-TDR-62-135

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Life Support Systems Laboratory  
6570th Aerospace Medical Research Laboratories  
Aerospace Medical Division  
Air Force Systems Command  
Wright-Patterson Air Force Base, Ohio

Contract Monitor: Clyde G. Roach  
Project No. 6373, Task No. 637302

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## FOREWORD

This report is a detailed summary of all work done on the subject contract. This work was performed at Isomet Corporation, Palisades Park, New Jersey, during the period from March 1, 1961, to February 28, 1962. The work was performed by Dr. Horace W. Chandler, Eugene McDonald, Frank Pollara, and Dr. George Walden under Contract No. AF33(616)-7909 in support of Project No. 6373, "Equipment For Life Support in Aerospace," Task No. 637302, "Respiratory Support Equipment."

Many helpful suggestions were contributed to this work by Professor T. I. Taylor of Columbia University, who served as a consultant on this project. Clyde G. Roach, Respiratory Equipment Section, Sustenance Branch, Life Support Systems Laboratory, served as contract monitor.

## ABSTRACT

On the basis of a survey the two most potentially valuable regenerable carbon dioxide sorption systems, molecular sieves and silver oxide preparations, were selected for laboratory investigation.

The 5A molecular sieve removed carbon dioxide from air containing 1 volume percent carbon dioxide, provided that water vapor was first removed from the air. At a flow rate of 500 cc/min. of this air through a 1-inch diameter column containing 100 grams of 5A molecular sieve, the bed picked up about 3 grams of carbon dioxide before carbon dioxide was detectable in the column effluent. Under identical operating conditions, a 100-gram bed containing a silver oxide preparation picked up about 8 or 9 grams of carbon dioxide. Water vapor in the air is necessary for efficient utilization of the silver oxide. Both the 5A molecular sieve and the silver oxide preparation are regenerable, but regeneration of the silver oxide occurs at a lower temperature.

## PUBLICATION REVIEW

This technical documentary report has been reviewed and is approved.

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## I. INTRODUCTION

An integral part of any closed-cycle life support system is a device or subsystem that converts exhaled carbon dioxide to oxygen suitable for breathing purposes. In order to accomplish this conversion, the carbon dioxide must first be removed from the air and then concentrated to a stream of pure carbon dioxide.

Many systems for carbon dioxide separation and recovery have been evaluated, either by reference to the literature or experimentally. Most of these incorporate one of the following procedures:

1. Freezing out by compression and cooling.
2. Absorption by amines or amino acid salts.
3. Absorption by alkali carbonates.
4. Absorption in sodium hydroxide from electrolysis of sodium sulfate.
5. Absorption in water or organic solvents.
6. Absorption by polyamine anion exchange resins.
7. Diffusion through semipermeable membranes.
8. Photosynthetic or photochemical methods.
9. Adsorption by solids such as molecular sieves.
10. Reactions with metal oxides such as silver oxide.

Based on an evaluation of these processes, the following conclusions have been reached:

1. Because of the high pressures and low temperatures required, the equipment necessary for a freezing-out process would be complex and the energy requirements high. It does not appear that such a process would be feasible with the severe limitations imposed by the requirements for low weight, low volume, and low power.

2. Absorption of carbon dioxide in various alkanolamine solutions is a known industrial process. The air emerging from this process, however, is contaminated with the amine vapors which are both disagreeable and toxic.

While the vapors can be removed, the increased complexity of the equipment and the mechanical and heat energy required for this process make it undesirable from the standpoint of carbon dioxide removal from closed space cabins.

3. Absorption of carbon dioxide in alkali carbonate solutions, although a known process of proven industrial utility, has many of the disadvantages cited in the previous process. It too requires the design of a gas-liquid contacting unit that will operate in a zero gravitational field.

4. Absorption of  $\text{CO}_2$  in NaOH solution is again a well-known process but has the disadvantages cited previously for gas-liquid absorption systems in zero gravitational fields.

5. The solubility of carbon dioxide in water and in organic solvents other than the ethanolamines is low, so that both the power requirements and the size of the equipment necessary to effect a given carbon dioxide separation would be greater than that required in other gas-liquid absorption processes.

6. The ability of polymeric amines to absorb carbon dioxide reversibly has been demonstrated previously by Gregor, (ref. 1). Although resin capacities appear to be adequate, the rates of absorption are low so that the size of the equipment becomes excessive.

7. The use of a membrane permeation process does not appear to be feasible because of the lack of selectivity of any known membrane for  $\text{CO}_2$ . In addition, low flow rates through the membranes will require excessive membrane area, thereby making the process both technically and economically unsound.

8. Photosynthetic methods utilizing algae and photochemical methods have not yet been developed to the point where they are technologically feasible under the conditions to be met in a space cabin.

9. Adsorption of carbon dioxide on solids such as molecular sieves appears to be a process which would be unaffected by zero-gravitational fields. The energy requirements of such a process could be small and the design of the equipment could be relatively straightforward. This process appears to be one of great potential utility.

10. Without further study, the ultimate utility of the absorption of carbon dioxide by silver oxide is not known. There appears, however, to be sufficient promise in this process, because of its simplicity and low energy requirements to warrant further detailed investigation.

The latter two methods, 9 and 10, appeared to be the ones of greatest potential utility. Accordingly our efforts were directed toward investigating the operating characteristics of these two systems.

## II. BACKGROUND INFORMATION

### Molecular Sieves

As mentioned above, one of the most useful materials in the class of solid adsorbents has been the class of materials known as molecular sieves. These materials, consisting of synthetic aluminosilicates, are produced with controlled pore sizes of molecular dimensions, and have the ability to adsorb certain gases and reject others. Although these materials are made under closely controlled conditions and the properties are quite similar from batch to batch, there are also small variations in properties from batch to batch.

The molecular sieve is available in the form of uniformly sized pellets which have good abrasion resistance. Contact with liquid water results in the development of considerable heat causing some decrepitation of the material. As has been pointed out by others, the capacity of the preparation for a given adsorbate depends on the chemical and physical properties of the adsorbent and adsorbate, the concentration of the adsorbate in the gas, and the temperature, pressure and flow rate used.

After sorption of the gases, molecular sieves can be regenerated either by heating, application of a vacuum, use of a purge gas or combinations of these techniques. The use of fixed beds of this material for CO<sub>2</sub> adsorption from air containing 1% by volume of CO<sub>2</sub> has been studied extensively both at Isomet and elsewhere.

Although molecular sieves were probably the best materials available for use in sorbing carbon dioxide from air prior to the start of this program, there are sufficient drawbacks to its use to make the search for newer and better carbon dioxide sorbers worthwhile. These drawbacks can be listed as follows:

1. Sensitivity to moisture. The presence of moisture or humidity in the inlet stream decreases the adsorption of carbon dioxide by the molecular sieve and, in some cases, may completely prevent it, the water being adsorbed in preference to the carbon dioxide.

2. Adsorption of other components in addition to CO<sub>2</sub>. In particular, the adsorption of N<sub>2</sub> by the molecular sieve and its subsequent desorption along with the carbon dioxide can lead to complications in the subsequent processing of the carbon dioxide. In at least one process, the CO<sub>2</sub> is converted chemically to carbon and O<sub>2</sub>. Introduction of N<sub>2</sub> into the process along with CO<sub>2</sub> can eventually lead either to marked dilution in

a process stream being used for the chemical conversion of the  $\text{CO}_2$  to  $\text{O}_2$  or to an increase in pressure inside the system, since the  $\text{N}_2$  would continue to build up in the system. It would, therefore, be necessary to have a bleed stream to maintain the nitrogen at some convenient level, and this bleed stream would represent inefficiency in the system.

3. High temperatures are required to regenerate the molecular sieve and this represents a net energy consumption by the process.

4. At the low partial pressure of  $\text{CO}_2$  in an air stream containing 1% by volume of carbon dioxide, the capacity of the molecular sieve is relatively low and, therefore, a large amount of this material would have to be carried in order to be able to run with reasonable adsorption and desorption cycles.

### Silver Oxide

Because of the problems inherent in the use of molecular sieve, as described above, there has been a continual search for new materials capable of reversibly sorbing and desorbing carbon dioxide. One of the most potentially promising materials in this respect has been silver oxide. The use of silver oxide as a reversible  $\text{CO}_2$  sorber was first studied in the 1940's as part of a program sponsored by the U. S. Navy. Results of this work indicated that  $\text{Ag}_2\text{O}$  could be used as a reversible  $\text{CO}_2$  sorber, but only if the  $\text{Ag}_2\text{O}$  was present in a form that displayed a large amount of surface. It appeared that the reaction of  $\text{CO}_2$  with the  $\text{Ag}_2\text{O}$  was limited by the rate of diffusion of the  $\text{CO}_2$  gas through the  $\text{Ag}_2\text{CO}_3$  surface layer on the  $\text{Ag}_2\text{O}$  particles.

The technique used in the study sponsored by the Navy overcame this handicap by distributing the silver oxide over the surface of a porous solid substrate. In this particular case, this was accomplished by soaking an activated alumina substrate in a caustic solution, rinsing, and then soaking the alumina in a silver nitrate solution, thereby precipitating the  $\text{Ag}_2\text{O}$  on the surface of the alumina. After drying, this material was found to absorb  $\text{CO}_2$  but it was never conclusively proved that this material could be regenerated completely since there was a marked and severe decline in  $\text{CO}_2$  absorbing ability after regeneration.

A marked disadvantage of this system was the low capacity for  $\text{CO}_2$  absorption. Only a small amount of active  $\text{Ag}_2\text{O}$  could be deposited by this technique, thereby necessitating the use of large weights of essentially inert carrier material. A further disadvantage of this system was the difficulty in regeneration. Heating to too high a temperature caused decomposition of the

$\text{Ag}_2\text{O}$  formed by decomposition of the  $\text{Ag}_2\text{CO}_3$ , thereby lowering the capacity of the system subsequent to regeneration.

In spite of these disadvantages, it was recognized that a system using silver oxide could be of great value if a way were found to overcome the difficulties discussed above, in particular the low capacity of the system. Although it was realized that in order to increase the capacity of the system, more  $\text{Ag}_2\text{O}$  having a high surface area would have to be incorporated in the system, this objective was not realized through the use of  $\text{NaOH}$  as a precipitant of the silver oxide. However, a more recent study at Isomet indicates that it is possible to prepare silver oxide in such a form that it has both a high capacity for carbon dioxide, and a high rate of carbon dioxide absorption. In order to accomplish this, silver carbonate is prepared in a very fine, microporous form by coprecipitating with another oxide which has a highly gelatinous structure in the wet form, but which becomes highly porous upon drying. The silver carbonate is converted to the oxide upon drying of the preparation under vacuum. The other oxide by its presence, serves to disperse the silver oxide in a very fine form and to maintain the preparation in a porous condition so that the gas is freely accessible to all portions of the preparation. In addition, by varying the proportions of silver oxide and the other oxide, the mechanical strength of the preparation can be changed from a rather friable, easily powdered material to a hard, strong material which makes the preparation suitable to a variety of uses.

The precipitation of the silver as a carbonate and decomposition to the oxide yields markedly better results than those obtained when the silver is precipitated directly as the oxide. This is probably because the silver oxide has been preconditioned by originally being in the carbonate form and the structure of the silver oxide has been adjusted to the absorption of carbon dioxide.

In these latter studies the  $\text{CO}_2$ -air stream was saturated with water and there appears to be no doubt that the presence of water played an important part in the markedly superior results obtained with the silver oxide. This is in contrast to the effect of water vapor on molecular sieves wherein water vapor is extremely detrimental to the operation. This constitutes one of the advantages of the silver oxide system over the molecular sieve system.

The method of preparation of the coprecipitate of silver carbonate with the other oxide is of great importance. We have found that coprecipitation of the silver carbonate with aluminum hydroxide yields a preparation that gives high carbon dioxide absorption rates and high carbon dioxide capacities, the capacity being

equivalent to the amount of silver in the preparation. Other oxides that might be used in place of the aluminum hydroxide are silicon dioxide, ferric hydroxide, and any other oxides that precipitate in a gelatinous form and dry to a highly porous mass.

The details of the method of preparation and of results obtained with these preparations are given in a later section.

### III. THEORETICAL CONSIDERATIONS

Two approaches have been used in the design of and interpretation of data obtained with fixed bed, gas absorption systems. In one, equations are obtained relating flow rate, column length, breakthrough time and concentrations to one another. In the other, the Mass Transfer Zone concept, which requires a knowledge of both the equilibrium capacity of the absorbent and the shape of the effluent concentration versus time curve, is used. We present both methods and will use both for interpretation of selected data.

#### Analytical Concept

The general equation describing the transfer of material from a gas to a stationary phase through which the gas is passing is (ref. 2):

$$-\frac{\partial C}{\partial a} = \frac{1}{L} \left( \frac{\partial x}{\partial T} + \frac{\partial C}{\partial T} \right) \quad (1)$$

- where T = total time that column has been exposed to absorbate  
 C = concentration of absorbate in gas at a point in the column  
 C<sub>o</sub> = concentration of absorbate in gas at the inlet to the column  
 L = linear flow rate of the gas stream  
 a = distance along the column from inlet end  
 x = amount of absorbate subjected to the absorption, per unit volume of absorbent.

In order to obtain a solution to this equation, an expression for  $\frac{\partial x}{\partial T}$ , the rate of absorption, must be used. We assume that the process takes place irreversibly and that the rate is proportional to the concentration of the absorbate in the gas and to the number of unreacted sites remaining on the absorbent.

$$\frac{\partial x}{\partial T} = kC (N_o - x) \quad (2)$$

- where N<sub>o</sub> = saturation capacity of the absorbent  
 k = mass transfer rate constant.

Substituting equation 2 into equation 1 and solving, we obtain

$$\ln \left( \frac{C_o}{C} - 1 \right) = kC_o T + \ln \left( \frac{kN_o \lambda}{L} - 1 \right) \quad (3)$$

where  $\lambda$  = column length.

If the assumptions are correct, a plot of  $\ln\left(\frac{C_0}{C} - 1\right)$  versus  $T$  should be linear, and the value of  $k$ , the mass transfer rate constant can be calculated from the slope of this line. It should be noted that the linear velocity,  $L$ , does not appear in equation 2 since it is assumed that the surface reaction is the rate-controlling step. In equation 3, therefore,  $L$  appears only as the term  $\frac{\lambda}{L}$ .

If the breakthrough time,  $t$ , is defined as the time at which the effluent concentration reaches a specified value  $C'$ , then the breakthrough time can be represented by the equation.

$$t + t_o = \frac{N_o \lambda}{C_o L} \quad (4)$$

$$\text{where } t_o = \frac{1}{kC_o} \left( \ln \left[ \frac{C_o}{C'} - 1 \right] \right)$$

It is seen that the breakthrough time depends only on  $\frac{\lambda}{L}$ .

If, however, diffusion is a controlling step in the process, then  $k$ , the mass transfer constant will be dependent on the flow rate and the breakthrough time will be dependent on  $L$  as well as on  $\frac{\lambda}{L}$  and plots of  $t$  versus  $\frac{\lambda}{L}$  will give different curves for different flow rates. This dependence on flow rates serves to indicate the presence of a slow diffusion step and if this does occur it is found that both  $k$  and  $N_o$  vary with flow rate.

From equation 4 we can obtain an expression for the critical volume,  $W_c$ , required.

$$W_c = \frac{V}{kN_o} \left( \ln \left[ \frac{C}{C'} - 1 \right] \right) \quad (5)$$

where  $W_c$  = critical bed volume  
 $V^c$  = volume of gas per unit time

Treatment of the volume of gas,  $V$ , per unit time, with the absorption of the quantity  $C_o V$  per unit time, requires the use of a minimum volume of absorbent,  $W_c$ . Quantities of absorbent less than this will not perform the desired removal.

If the rate of absorption is limited by the reaction between the absorbate and the absorbent, then low flow rates would yield better results. If diffusion of the absorbate from the gas stream to the surface of the absorbent is the limiting step, then high flow rates will be desirable. As will be seen later, both rate-limiting steps are important when silver oxide is used as an absorbent. When the silver oxide is fresh, the diffusion of  $CO_2$  from the gas stream to the absorbent surface is the



limiting step. After the particle surface has been converted from  $\text{Ag}_2\text{O}$  to  $\text{Ag}_2\text{CO}_3$ , the rate of absorption becomes dependent on the rate of diffusion of the  $\text{CO}_2$  through the surface layer of  $\text{Ag}_2\text{CO}_3$  to the underlying layer of  $\text{Ag}_2\text{O}$ .

### Mass Transfer Zone Concept

Michaels (ref. 3) has developed the Mass Transfer Zone concept that is limited to sorption from dilute feed mixtures and to cases where the sorption zone is constant in height as it travels through the sorption column and where the bed height is large relative to the height of the sorption zone. The use of this method is also restricted to those cases where the sorption equilibrium isotherm is concave to the solution-concentration axis. The sorption zone is that portion of the bed in which the concentration of sorbate in the gas changes from its value at the saturated front portion of the bed to the value of the effluent concentration.

Consider the flow of  $G$  lb./hr.ft.<sup>2</sup> of solvent gas having an initial solute concentration of  $Y_0$  lb. solute/lb. solvent gas through the sorbing bed. The breakthrough concentration is defined as  $Y_B$  and the effluent concentration when the bed is essentially exhausted is defined as  $Y_E$ .  $w_B$  and  $w_E$  are defined as the quantity of solute-free gas that has passed through the column at breakthrough and at saturation. An idealized breakthrough curve is shown in Figure 1. The total effluent collected during the breakthrough period is  $w_A = w_E - w_B$ . The Mass Transfer Zone,  $Z_a$ , is that portion of the bed in which the concentration of the sorbed gas changes from  $Y_B$  to  $Y_E$ .

It has been shown that  $Z_a$  can be calculated from the following equation:

$$Z_a = Z \frac{\theta_A}{\theta_E - (1-f)\theta_A} = Z \frac{w_A}{w_E - (1-f)w_A} \quad (6)$$

Where  $Z$  = column height,  
 $\theta_A$  = time required for Mass Transfer Zone to move its own height,  
 $\theta_E$  = time for Mass Transfer Zone to establish itself and move out of the bed,  
 $f = U/Y_0 w_A$  = fractional ability of the sorbing material in the Mass Transfer Zone still to sorb solute at breakthrough point.

It is clear that a good sorber will have a high equilibrium capacity and a short Mass Transfer Zone. A knowledge of the Mass Transfer Zone Length and the equilibrium data for the

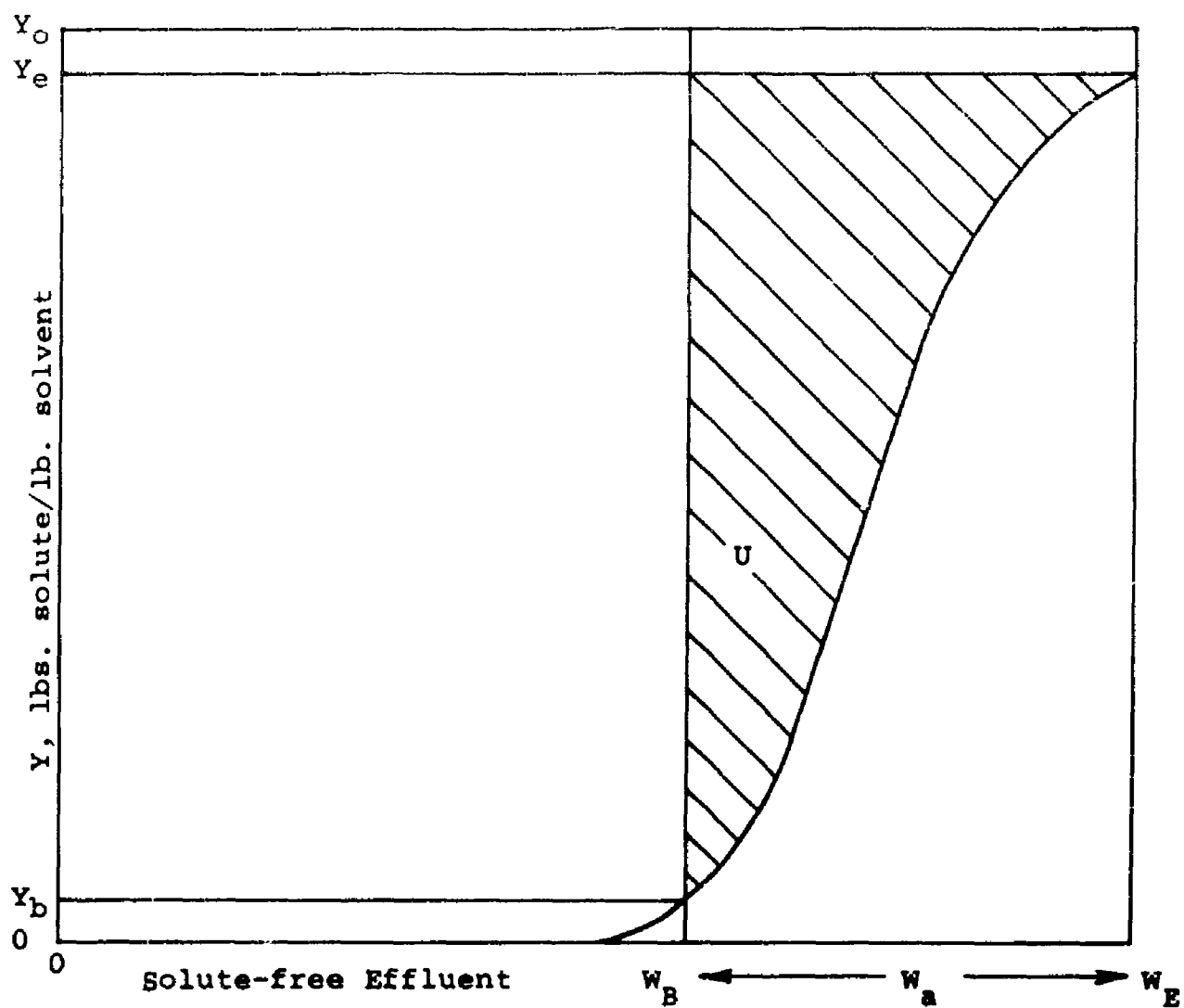


Figure 1. Breakthrough Time Profile

sorber combined with information regarding cycle time, desorption time, pressure drop and gas flow rate requirements allows the design of the CO<sub>2</sub> sorption system.

#### IV. EXPERIMENTAL PROGRAM

As mentioned in the Introduction, the two systems chosen for further study were those based on molecular sieve 5A and on silver oxide. In general, the procedures used for study of the sorbers were similar for all sorbers. Air containing  $\text{CO}_2$  at various concentrations was passed at different flow rates under conditions of varying temperature and humidity through packed beds of the various sorbents and the inlet and outlet concentration of  $\text{CO}_2$  in the gas streams monitored over the period of the run. Details of the various experimental procedures are given below.

##### Molecular Sieve

The columns were made of 1" I.D. glass pipe of varying lengths. Approximately 100 grams of 1/8" Linde 5A molecular sieve pellets were poured into the columns. The columns were equipped with thermocouple wells and were wrapped with heating tape for the desorption operation described below. Suitable tubing, thermocouples, flowmeters and pumps were provided.

The air- $\text{CO}_2$  mixtures were obtained from cylinders made up to the desired concentration by the Matheson Company. The concentration of the  $\text{CO}_2$  in these gases was checked by calibration against pure  $\text{CO}_2$ . The  $\text{CO}_2$  concentrations in the inlet and outlet gas streams were determined using a gas chromatograph capable of measuring concentrations of 0.1%  $\text{CO}_2$ . In each run, dry air containing 1 percent  $\text{CO}_2$  by volume was run through the molecular sieve at a given flow rate until the concentration of carbon dioxide in the effluent arrived at some predetermined fraction of that of the feed stream.

In those studies where it was desired to reuse a column of adsorbent, the material was regenerated by heating and applying a partial vacuum. Prior to heating, the column was evacuated for several minutes to remove gases other than  $\text{CO}_2$  that were in the column but not necessarily adsorbed. Two methods were used to determine the amount of  $\text{CO}_2$  desorbed. In the first, the column was weighed directly before and after regeneration. In the second, the desorbed  $\text{CO}_2$  was collected on an ascarite-molecular sieve mixture and the gain in weight of the mixture determined. Molecular sieve was mixed with the Ascarite since it was noticed that the Ascarite alone soon plugged when the desorbed  $\text{CO}_2$  collected on the Ascarite. The time, temperature and pressure used during the regeneration process were varied.

A series of runs was made to determine the effect of temperature on the desorption of carbon dioxide from the molecular sieve.

A single charge was used for these runs. The column was weighed after each desorption period and the total cumulative weight loss, based on the initial weight of the column and its adsorbed carbon dioxide, was noted. The desorption was carried out for about 6 hours at a pressure of 3 mm. Hg.

A series of desorption runs was carried out at 250°C with a pressure of 28 cms. Hg. This vacuum was used because it represents what can be obtained with a light-weight, compact vacuum pump capable of operating under zero gravity conditions. As will be seen later, the pressure was later lowered to 3 mm. Hg. to improve the regeneration.

The stability of the molecular sieve was determined in a series of runs. The apparatus used for adsorption of carbon dioxide on molecular sieve is essentially the same as described previously. Analyses were carried out by absorption of carbon dioxide on either Ascarite or Indicarb. Two columns, each containing 100 grams of 1/8-inch 5A molecular sieve pellets were used in order to determine reproducibility of the data. The feed gas was 1% carbon dioxide in dry air flowing at a rate of 3 liters per minute. Immediately before breakthrough occurred, as determined by preliminary experiments, tubes containing a carbon dioxide absorber were placed in the stream from the molecular sieve column. These tubes were changed every 10 minutes for a period of 40 minutes. Exact breakthrough time was established by plotting cumulative weight of carbon dioxide back to zero carbon dioxide in the absorber tubes. The air flow rate was adjusted using a rotameter and the exact feed rate measured by passing the feed stream through the absorbing tubes for a measured time after a run was completed. Carbon dioxide adsorbed on the molecular sieve was calculated using the measured feed rate.

After the first three runs in each series, regenerating was carried out at 250°C and 3 mm. Hg. rather than 250°C and 28 cms. Hg. Regeneration was carried out overnight in both cases.

### Silver Oxide

#### a. Silver Oxide-Silica Gel Mixture

Commercial "water glass" was used in the preparation of the  $\text{Ag}_2\text{O-SiO}_2$  mixtures. The water glass was precipitated from solution using sulfuric acid. A known amount of  $\text{AgNO}_3$  solution was added to the gel, followed by  $\text{Na}_2\text{CO}_3$ , to precipitate the  $\text{Ag}_2\text{CO}_3$ . The mixture was stirred thoroughly, decanted, and dried at 120°C and 1 atm. The charge was then heated to 175°C under vacuum to decompose the  $\text{Ag}_2\text{CO}_3$  to  $\text{Ag}_2\text{O}$ .

b. Pure Silver Oxide

Pure silver oxide was prepared by precipitation from a silver nitrate solution using NaOH as the precipitant. The precipitate was filtered, washed and finally dried at 125°C and 1 atm. The charge was placed in a glass column and a stream of 3% CO<sub>2</sub> in N<sub>2</sub> was fed through it.

c. Silver Oxide-Aluminum Oxide Mixture

Silver oxide and aluminum oxide were precipitated from a AgNO<sub>3</sub>-Al(NO<sub>3</sub>)<sub>3</sub> solution by raising the pH of the solution to approximately 8 using a NaOH solution. The precipitate was filtered and washed, but drying was done under varying conditions depending on the particular run.

d. Silver Oxide on Bauxite

Silver oxide was deposited on bauxite by the following procedure. The activated bauxite, in a stainless steel basket was allowed to soak in a solution of 25% NaOH for 10 minutes, rinsed briefly in distilled water, placed in a 25% AgNO<sub>3</sub> solution, agitated for 5 minutes and rinsed again. The charge was drained and dried (except where noted) at 125°C and 1 atm. for four hours and activated at 125°C and 3 mm. Hg. overnight.

Where indicated, the Ag<sub>2</sub>O- bauxite charge was wetted by passing through it a stream of O<sub>2</sub> saturated with water at room temperature. In all cases where a wet or rewetted charge was used, the feed stream was saturated with water also. The amount of silver on the bauxite preparations was determined via Volhard titrations. The necessary blanks and checks were performed using this procedure to insure reliability of the analyses.

e. Silver Oxide on Pumice

Silver oxide was deposited on 10/20 mesh pumice using the technique described under (d) with one modification. The rinsing in distilled water between the NaOH bath and the AgNO<sub>3</sub> solution was limited to 1-2 seconds using only one bath. As shown in Table 5, the amount of Ag<sub>2</sub>O deposited on this substrate was considerably greater than that deposited on the activated bauxite. Batch 2, used in Run 1, was dried at 125°C in air for 48 hours, whereas Batch 3 used in Run 2 was dried at 125°C in air for only 16 hours. In both cases, the feed stream was water-saturated air containing 1% CO<sub>2</sub> at 27°C flowing at 500 CC/min.

f. Silver Oxide on 5A Molecular Sieve

5A molecular sieve (100 grams) was first saturated slowly with water to avoid the generation of large amounts of heat which would crack and spall the molecular sieve. This material was then soaked in a sodium hydroxide solution (25% by weight) for 15 minutes, rinsed quickly in 3 separate distilled water baths and then immersed in a 25% solution of  $\text{AgNO}_3$  for 5 minutes with agitation. The silver oxide precipitated on the molecular sieve. The resulting preparation was filtered, air dried at  $115^\circ\text{C}$  and 1 atm. pressure, vacuum dried at  $150^\circ\text{C}$  and 3 mm. Hg. pressure and then dried further at  $250^\circ\text{C}$  in an oxygen stream in an effort to remove more water from the molecular sieve.

g. Silver Oxide on Asbestos

A mixture of  $\text{AgNO}_3$ ,  $\text{Al}(\text{NO}_3)_3$  and  $\text{NaOH}$  containing a 1:1:4 ratio of  $\text{Ag}^+1$ : $\text{Al}^{+3}$ : $\text{OH}^{-1}$  was deposited on asbestos fiber, washed and dried, and activated at  $125^\circ\text{C}$  and 3 mm. Hg. for 16 hours.

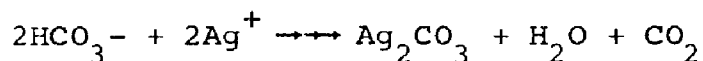
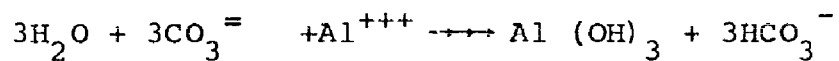
h. Silver Oxide prepared by  $\text{NH}_4\text{OH}$  Precipitation

In an effort to eliminate any effect of excess  $\text{NaOH}$  in the silver oxide preparation, silver carbonate was dissolved in concentrated  $\text{NH}_4\text{OH}$  and poured onto bauxite. The charge was heated to drive off  $\text{NH}_3$  and  $\text{H}_2\text{O}$  and activated at  $125^\circ\text{C}$  and 3 mm. Hg. for 16 hours. Silver oxide was also deposited on asbestos fiber with a similar technique using concentrated  $\text{NH}_4\text{OH}$  and  $\text{AgNO}_3$ .

This procedure should be carried out with extreme care since silver fulminate ( $\text{AgN}_3$ ) may form and decompose explosively on heating.

i. Silver Oxide-Aluminum Oxide Mixture by  $\text{Ag}_2\text{CO}_3$  Precipitation

Mixtures of silver oxide and aluminum oxide (partially hydrated) were prepared in the following way.  $\text{AgNO}_3$  and  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  were dissolved in approximately 1500 cc. of water in the desired ratio. An amount of  $\text{Na}_2\text{CO}_3$  sufficient to satisfy the equations below was dissolved in a second 1500 cc. of water. The solution containing the silver and aluminum nitrates was added as rapidly as possible without causing excessive frothing to the sodium carbonate solution. The silver precipitated as silver carbonate and the aluminum as aluminum hydroxide according to the following equation:



The precipitate was filtered on a Buchner funnel and then suspended in a water solution. This process was followed twice more in order to wash out any excess sodium carbonate. The final wet precipitate was then extruded through a 10-mesh nickel screen and dried at 120°C and 1 atmosphere in air for 16 hours.

Following the drying, the silver carbonate was decomposed to silver oxide by heating the mixture to 170-175°C for 16 hours at 2 mm. Hg. pressure followed by treatment at 200°C for 6-9 hours at 2 mm. Hg. pressure. The following table indicates the amounts of reagents required to make approximately 100 grams of final dry mixture.

<u>Ag<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub></u>	<u>Na<sub>2</sub>CO<sub>3</sub>, gms.</u>	<u>Al(NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O, gms.</u>	<u>AgNO<sub>3</sub>, gms.</u>
80/20	125	147	117.5
50/50	312	367	73.2
20/80	513	595	29.3

Approximately equal weights of these preparations were placed in three separate columns and air saturated with water and containing 1% by volume carbon dioxide was passed through the column at a rate of 500 cc./min. at room temperature. The concentration of the carbon dioxide in the effluent gas as a function of time is determined gas chromatographically.



## V. EXPERIMENTAL RESULTS AND DISCUSSION

### Molecular Sieve

Curves showing the variation of  $\text{CO}_2$  concentration in the effluent gas with the volume of gas handled for different flow rates and a fixed size bed of 5A molecular sieve are shown in Figure 2. As expected, the volume of gas that can be handled up to the breakthrough point decreases as the flow rate increases. The variation of  $\text{CO}_2$  loading at the breakthrough with flow rate is shown in Table 1 and Figure 3.

The results indicate that loading at breakthrough increases as the flow rate decreases. Both of these observations are in agreement with previous work on similar systems (refs. 4,5,6). It should be pointed out that not all of the decrease in capacity shown in Figure 3 is due to the increase in flow rate. A subsequent study at constant flow rate showed that 5A molecular sieve not previously used, exhibits a decrease in loading at breakthrough during the first few cycles of adsorption and regeneration. The extent of this decrease depends on the temperature and pressure used during the desorption process. A comparison of Runs A-2, B-2 and C-2, wherein the adsorbent was subjected to the same number of cycles and hence the same preconditioning, more truly reflects the effect of flow rate. It is seen that an increase in flow rate from 1.5 to 5 liters per minute results in a decrease in loading from 5.9 to 3.5 grams of  $\text{CO}_2$  per 100 grams of 5A molecular sieve. The latter result appears to be slightly higher than that obtained by other workers (ref. 6) under similar flow conditions.

Table 2 shows the effect of temperature on desorption of  $\text{CO}_2$  from 5A molecular sieve. As is expected, the amount of  $\text{CO}_2$  removed from the bed increases as the temperature rises. At  $250^\circ\text{C}$ , 92 percent of the  $\text{CO}_2$  was removed from approximately 100 grams of the molecular sieve.

The data in Figures 4 and 5 show the stability of 5A molecular sieve over a period of 15 adsorption and regeneration cycles. In this series of runs, two columns were used and subjected to identical conditions in order to determine the reproducibility of the data. The columns were not thermostated and the temperature during the regeneration cycle may have varied as much as  $\pm 10^\circ\text{C}$ . This was probably the main reason for the fluctuation in the data.

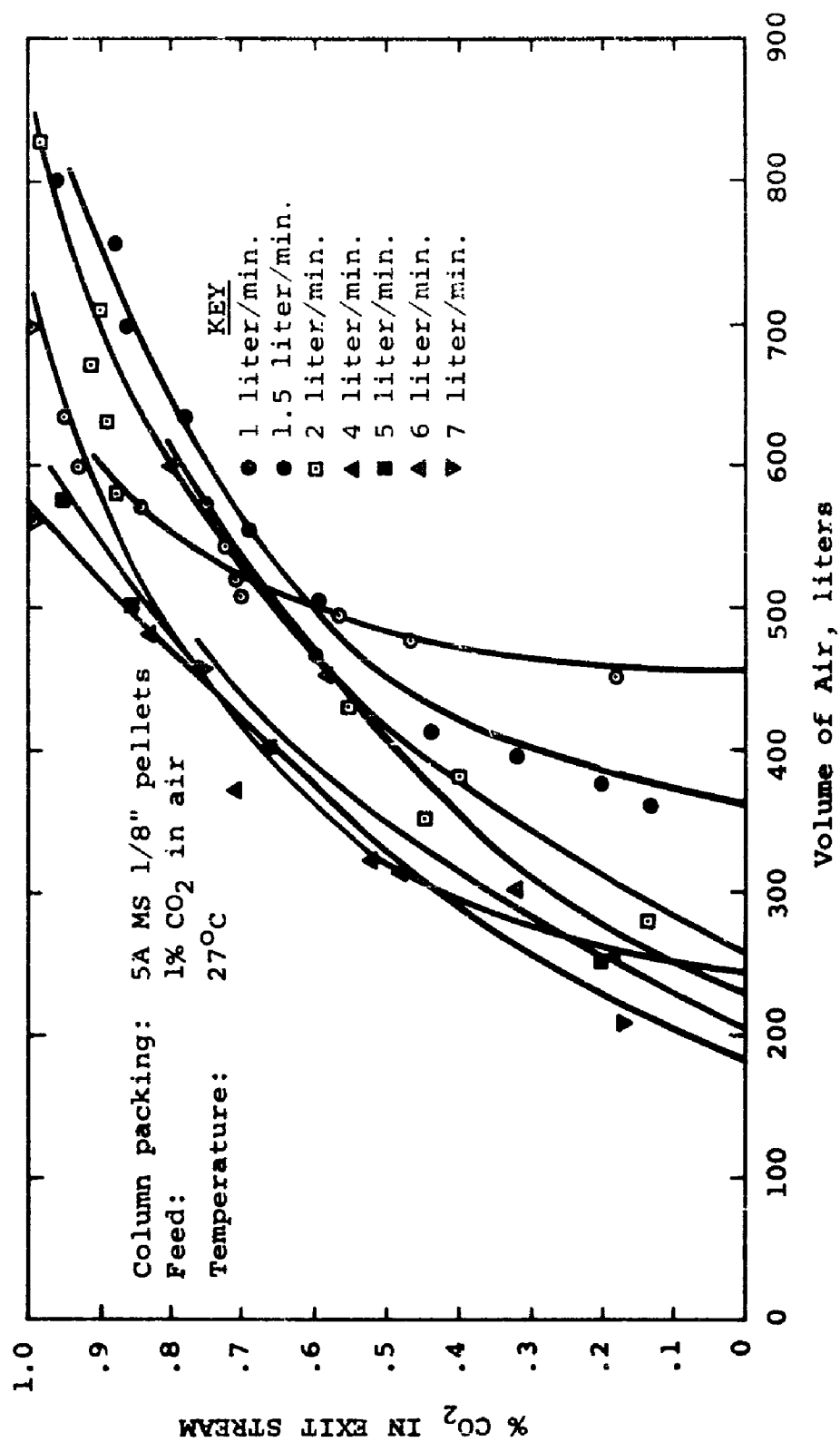


Figure 2. Variation of CO<sub>2</sub> in Effluent Gas with Volume of Gas Handled

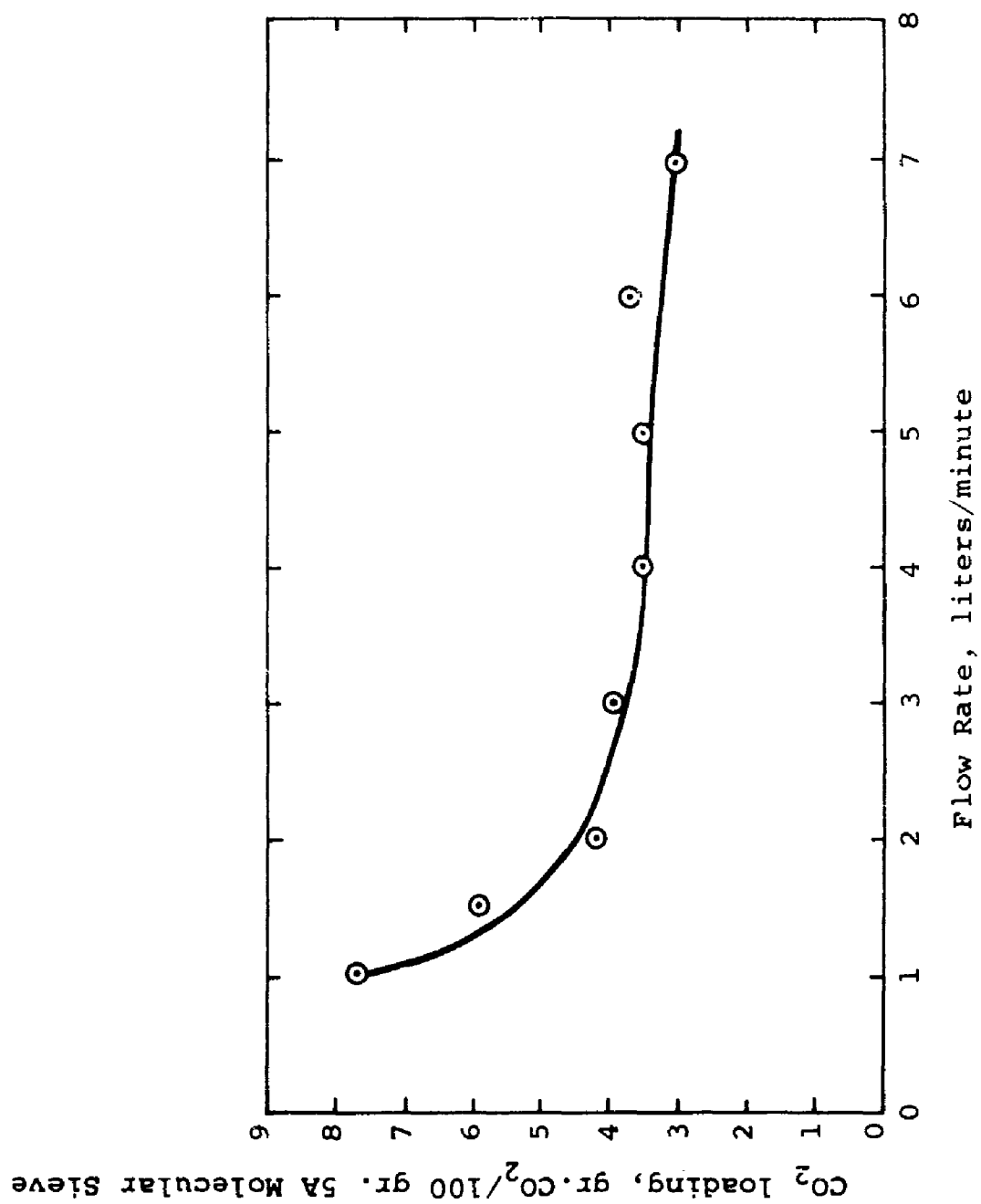


Figure 3. Effect of Flow Rate on CO<sub>2</sub> Loading at Breakthrough

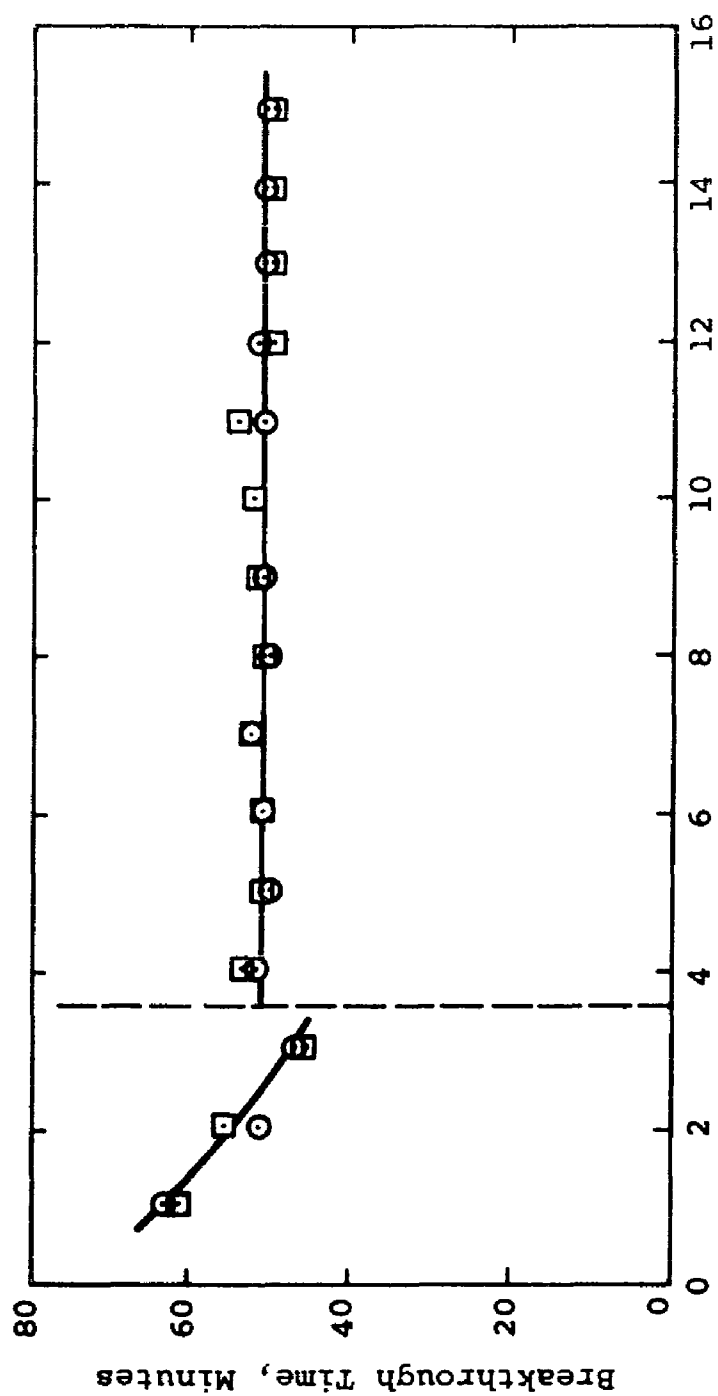


Figure 4. Cyclic Stability of 5A Molecular Sieve

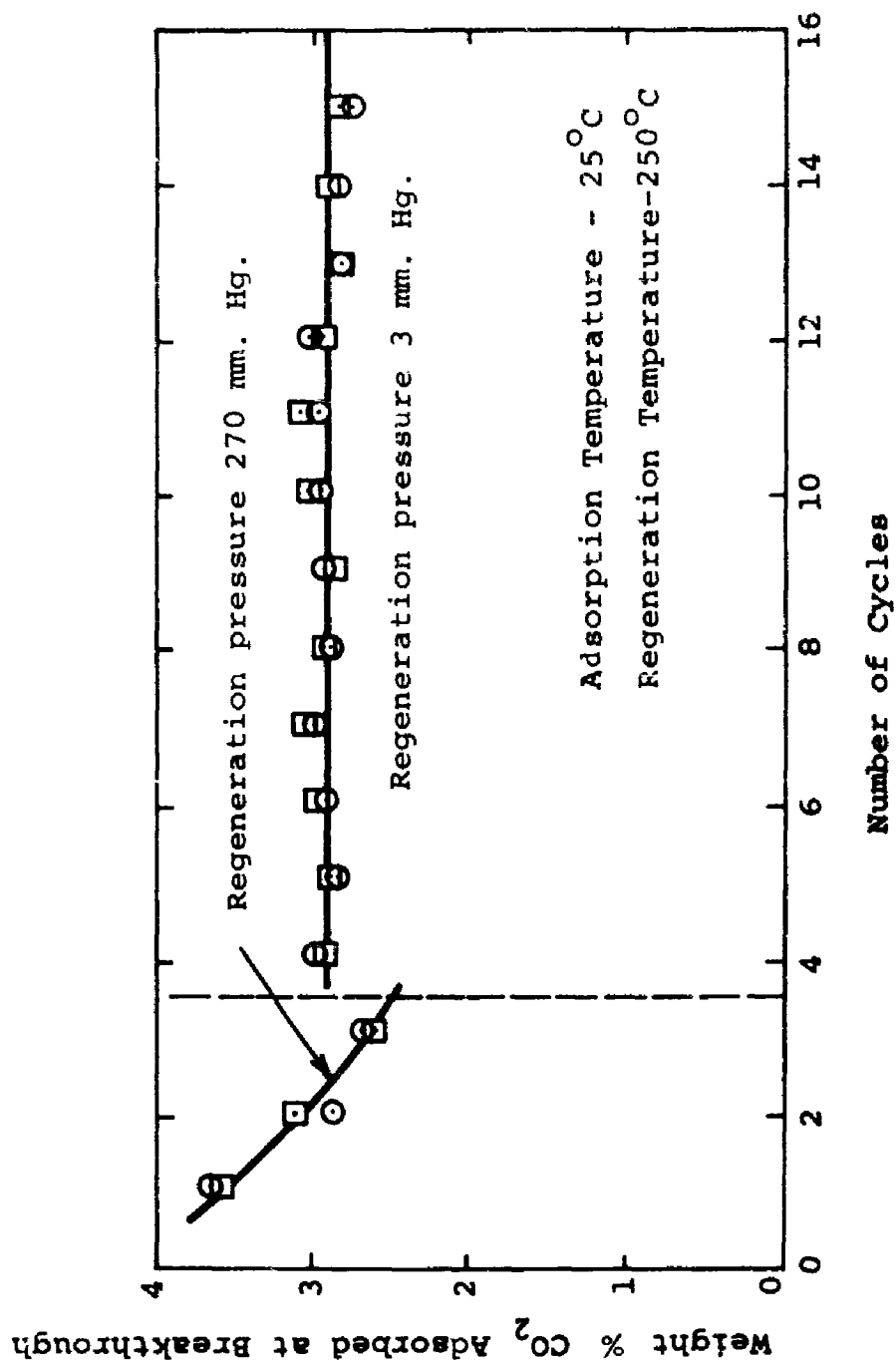


Figure 5. Cyclic Stability of 5A Molecular Sieve

TABLE 1

EFFECT OF FLOW RATE ON CO<sub>2</sub> LOADING AT BREAKTHROUGH

1/8-Inch Type 5A molecular sieve.

27°C

Column Diameter - 1 inch I.D.

Breakthrough

Loading, CO <sub>2</sub> /100 g.MS5A	(a) Time, Minutes	Flow Rate, liters/min.	Sieve Designation <sup>(b)</sup>		
			A	B	C
(c)	--	--	1		
7.7	440	1.0		1	
5.9	230	1.5	2		
4.2	120	2.0		2	
3.9	75	3.0			1
3.5	50	4.0		3	
3.5	40	5.0			2
3.7	35	6.0		4	
3.0	25	7.0			3

Notes: (a) Calculated

(b) The letters A, B and C designate the column charge while the numbers indicate the number of times the particular charge has been used.

(c) First run on charge A omitted because of variation in CO<sub>2</sub> concentration in feed stream.

Column A - 107.7 g.

Column B - 105.1 g.

Column C - 106.5 g.

TABLE 2

EFFECT OF TEMPERATURE ON DESORPTION OF CARBON DIOXIDE  
FROM 5A MOLECULAR SIEVE

Temperature, °C	Pressure, mm. Hg.	Desorption Time, hours	Cumulative % CO <sub>2</sub> Desorbed
25	3	6.5	44
60	3	6.5	77
100	3	5.0	87
150	3	6.5	86
250	3	6.5	92

Examination of the data indicated its excellent reproducibility. The capacity of the molecular sieve appears to be constant after an initial small decline. This initial decline can be explained by the fact that the desorption of the first 3 cycles was carried out at 270 mm. Hg. pressure, at which pressure desorption is not complete. On the next cycle, therefore, the capacity is reduced. The capacity of the molecular sieve would probably have leveled off at some lower value if the desorption step had continued to be carried out at 270 mm. Hg. When the desorption was carried out at 3 mm. Hg., after the first three cycles, the capacity remained constant.

Silver Oxide

a. Silver Oxide-Silica Gel Mixture

The experiments on the Ag<sub>2</sub>O-SiO<sub>2</sub> mixtures containing 50% Ag<sub>2</sub>O and prepared as described in Section IV, were carried out in a 3/4-inch I.D. glass column 12 inches long. A 30-gram charge of the Ag<sub>2</sub>O preparation was placed in the column and approximately 30 cc./min. of pure CO<sub>2</sub> was passed through the column at temperatures ranging from 25° to 125°C. A fresh charge from the same batch was used for each run. The column temperature was controlled with a heating tape wrapped around the column and the temperature monitored with a chromel-alumel thermocouple. Each run lasted 90 minutes with the Indicarb sampling tubes in the exit stream being changed every 15 minutes. The results of these experiments are shown in Table 3. Figure 6 shows the absorption versus time curves for operation at 25°, 75° and 125°C.

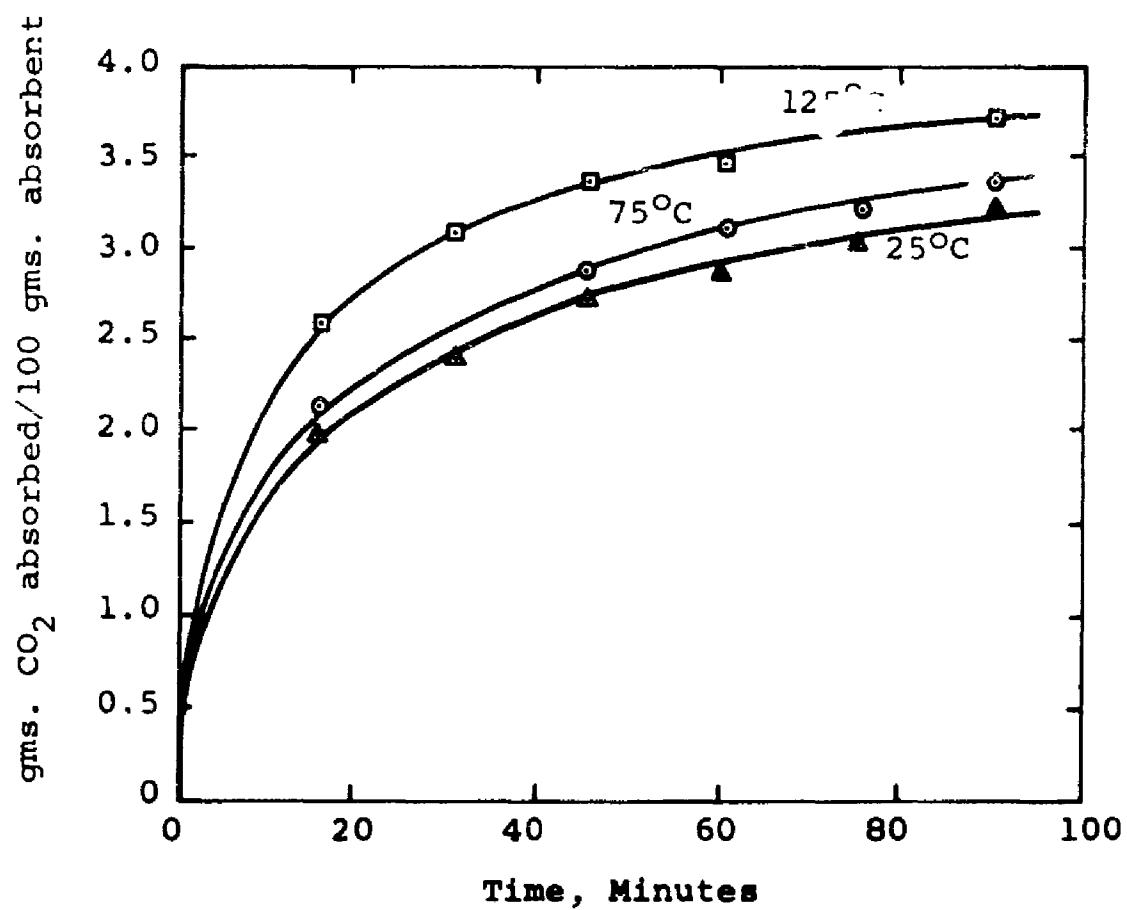


Figure 6. Absorption of CO<sub>2</sub> by Ag<sub>2</sub>O-SiO<sub>2</sub> (50/50) mixture



TABLE 3

ABSORPTION OF CO<sub>2</sub> ON Ag<sub>2</sub>O-SiO<sub>2</sub> MIXTURES

Run No.	Temp. °C.	Total CO <sub>2</sub> Absorbed gms.	gm. CO <sub>2</sub> 100 gm. Ag <sub>2</sub> O	Average Feed Breakthrough Rate(cc./min.)	Time(Min.)
7	25	0.98	6.53	29.8	10
8	50	1.105	7.36	30.8	10
9	75	1.024	6.84	30.0	10
10	100	1.161	7.75	30.4	10
11	125	1.123	7.50	30.5	10

The increase in CO<sub>2</sub> absorbed from 25°C to 125°C is approximately 15 percent. The curves in Figure 6, however, show that the major part of this increase occurred during the first 15 minutes while during the remaining 75 minutes, the rate of absorption was about the same in all cases. This is probably caused by the initial increase in reaction rate between the surface layers of Ag<sub>2</sub>O and the CO<sub>2</sub> as the temperature increases, while during the remainder of the run, diffusion of CO<sub>2</sub> to the underlying layers of Ag<sub>2</sub>O which is not as temperature dependent a process, becomes the rate controlling mechanism.

Preliminary experiments with air containing 1% CO<sub>2</sub> by volume indicated negligible absorption of CO<sub>2</sub> by this preparation. The experiments with pure CO<sub>2</sub> were performed in an effort to see whether any CO<sub>2</sub> could be absorbed under any conditions.

b. Pure Silver Oxide

The gas consisting of a mixture of 3 per cent CO<sub>2</sub> in nitrogen was fed through the pure Ag<sub>2</sub>O at 500 cc./min. Breakthrough was immediate and there was no detectable CO<sub>2</sub> absorption. At a flow rate of 110 cc./min. using the same feed composition, a total of 0.03 weight per cent CO<sub>2</sub> was picked up on an Ag<sub>2</sub>O charge after 35 minutes of absorption. In both cases the runs were carried out at room temperature. Results are shown in Table 4.

c. Silver Oxide-Aluminum Oxide Mixture

The results obtained with the Ag<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub> mixtures prepared by direct precipitation with NaOH as described in Section IV are shown in Table 4.

TABLE 4

CO<sub>2</sub> ABSORPTION BY Ag<sub>2</sub>O PREPARED BY DIRECT PRECIPITATION WITH NaOH

Run	Coprecipitant	Ag <sub>2</sub> O%	Gas Feed		Temp. °C	CO <sub>2</sub> Absorbed		Time Min.
			% CO <sub>2</sub>	Rate, cc./min.		Actual %	Theory %	
14	None	100	3.0 *	425	25	0	24	--
15	None	100	3.0*	110	25	0.03	24	35
16	Al <sub>2</sub> O <sub>3</sub>	76	1.0	250	25	0	14.4	--
17	Al <sub>2</sub> O <sub>3</sub>	70	1.0	250	25	0	13.2	--
18	Al <sub>2</sub> O <sub>3</sub>	60	1.0	1000	25	0	11.4	--
19	Al <sub>2</sub> O <sub>3</sub>	60	100	30	25	6.2	11.4	195

\* Nitrogen used as diluent

In Run 18, the charge was dried initially at 125°C and 1 atmosphere for 16 hours, then at 250°C in a stream of O<sub>2</sub> for 6 hours. For Run 16, drying took place at 125°C and 1 atmosphere whereas in 17, the precipitate was dried at 125°C and 3 mm. Hg.

d,e,f,g. Ag<sub>2</sub>O on Bauxite, Pumice, 5A Molecular Sieve and Asbestos

Data for Ag<sub>2</sub>O deposited on the various substrates are shown in Table 5. It will be noted that in all cases where the materials were carried through several cycles of absorption and regeneration, the capacity both at breakthrough and total, declined on each subsequent cycle. In all cases the amounts of CO<sub>2</sub> absorbed were very small and the capacities of these preparations for CO<sub>2</sub> at breakthrough were considerably below those obtained with 5A molecular sieve. It can be seen that in all cases except where asbestos was used, the amount of Ag<sub>2</sub>O incorporated in the preparation was very small.

In some cases, the amount of CO<sub>2</sub> sorbed on the material is more than can be accounted for by the amount of silver oxide present. This is probably caused by experimental variations due to the small amount of silver oxide present in the preparations. In the case where 5A molecular sieve was used as a substrate upon which the Ag<sub>2</sub>O was deposited, it is not known whether the CO<sub>2</sub> was being sorbed by the Ag<sub>2</sub>O or the molecular sieve. In any event, the CO<sub>2</sub> sorption was less than that obtained with molecular sieve alone.

h. Silver Oxide Prepared by NH<sub>4</sub>OH Precipitation

As shown in Table 6, none of the Ag<sub>2</sub>O preparations made by precipitation of the Ag<sub>2</sub>O with NH<sub>4</sub>OH exhibited any CO<sub>2</sub> absorption.

i. Silver Oxide-Aluminum Oxide by Ag<sub>2</sub>CO<sub>3</sub> Precipitation

Table 7 gives the results obtained with all silver oxide preparations made by decomposing silver carbonate. The absorption times given in this table represent a convenient time for run termination rather than 100% CO<sub>2</sub> saturation. In all cases, however, the CO<sub>2</sub> loading at termination of the run was greater than 90% of the maximum as determined by extrapolation of the absorption curves. The charge designated as 3 in Table 7 was used unregenerated in Run 4. The charge from Run 4, however, was regenerated at 175°C and 3 mm. Hg. and used in Run 5.

TABLE 5  
ABSORPTION OF CO<sub>2</sub> BY Ag<sub>2</sub>O DEPOSITED ON VARIOUS SUBSTRATES

Run	Substrate	Charge History (1)	Charge Composition % H <sub>2</sub> O % Ag <sub>2</sub> O	gr. CO <sub>2</sub> /100 gr. absorbent		Time (Min.)	Regeneration
				Experimental	Theoretical		
20	Bauxite	B1,S1,C1	0 2.7	.22 .34	.51	22 -	125°C, 3 mm Hg, 16 hours
		C2	0 2.7	.17 .32	.51	20 -	125°C, 3 mm Hg, 16 hours
		C3	0 2.7	.15 .27	.51	17 -	-
21	Bauxite	B1,S1,C1	0 2.6	.62 .68	.49	68 -	125°C, 3 mm Hg, 16 hours
		C2	0 2.6	.42 .52	.49	42 -	125°C, 3 mm Hg, 16 hours
		C3	0 2.6	.35 .44	.49	33 -	-
22	Bauxite	B2,S2,C1	5.9 2.4	.92 1.14	.49	104 -	125°C, 3 mm Hg, 16 hours
		C2	5.9 2.4	.76 0.78	.49	75 -	125°C, 3 mm Hg, 16 hours
		C3	5.9 2.4	.73 0.79	.49	69 -	-
23	Bauxite	B5,S1,C1	29.0 1.9	.80 2.90	.36	110 570	Steam, 1 atm., 125-150°C, 1 hr.
		C2	5.8 2.2	.24 .34	.42	25 90	-
24	Bauxite	B6,S1,C1	29.8 1.6	.45 1.78	.30	70 503	Sat. steam, 130-160°C, 1 hr.
		C2	- 2.2	0 .18	.42	- 191	-
25	Ms-5A	B1,S1,C1	- -	.58 1.35	-	32 235	None
26	Ms-5A	B2,S1,C1	- -	- 1.36	-	- 220	150°C, 3 mm Hg, 16 hours
		C2	- -	- .66	-	- 210	-
27	Asbestos	B2,S1,C1	17.5 20	1.02 3.43	4.55	125 500	125°C, 3 mm Hg, 16 hours
		C2	17.5 20	0 .55	4.55	- 125	-
1	Pumice	B2,S1,C1	31.0 6.4	.21 1.25	1.17	20 260	Sat. steam, 160°C, 4 hours
		C2	-	0 .08	-	0 125	-
2	Pumice	B3,S1,C1	0 12.7	0 2.6	2.4	0 1800	150°C, 2 mm Hg, for 2 hours
		C2	-	0 1.6	2.4	0 2100	-

(1) E.G. - Run 20 - Batch 1, Sample 1, Cycles 1, 2 and 3

Temp.: 27°C Rate: 500 cc./min. of 1% CO<sub>2</sub> in air

(2) Cut off at point where effluent was 90% of Feed concentration.

TABLE 6

ABSORPTION OF CO<sub>2</sub> BY Ag<sub>2</sub>O PREPARED WITH NH<sub>4</sub>OH

Run No.	Charge Treatment	Substrate	% Ag <sub>2</sub> O	% CO <sub>2</sub> Absorbed		Regeneration
				Actual	Theoretical	
28	BI, SI, CI	Asbestos	25	0	4.75	185°C, 3 mm.Hg. 16 hours
		Asbestos	--	0	4.75	--
29	B3, SI, CI	Bauxite	25	0	4.75	125°C, 3 mm.Hg., 16 hours
		Bauxite	--	0	4.75	--

TABLE 7

CO<sub>2</sub> ABSORPTION BY Ag<sub>2</sub>O PREPARED THROUGH THE CARBONATE

No.	Absorbent Composition		Feed Rate (cc/min.)	Column Temp. °C	Time Min.	% CO <sub>2</sub> Absorbed		(a)
	Co- precipitant	Nominal Ag <sub>2</sub> O %				Actual	Theoretical	
1.	SiO <sub>2</sub>	26	100	30	25	2.6	4.9	
2.	SiO <sub>2</sub>	38	100	30	25	1.3	7.2	
3.	SiO <sub>2</sub>	38	100	30	25	2.3	7.2	
4.	SiO <sub>2</sub>	38	100	30	50	2.7	7.2	
5.	SiO <sub>2</sub>	38	100	30	75	2.0	7.2	
6.	SiO <sub>2</sub>	50	100	30	25	3.3	9.5	
7.	SiO <sub>2</sub>	50	100	30	50	3.7	9.5	
8.	SiO <sub>2</sub>	50	100	30	75	3.4	9.5	
9.	SiO <sub>2</sub>	50	100	30	100	3.9	9.5	
10.	SiO <sub>2</sub>	50	100	30	125	3.8	9.5	
11.	Al <sub>2</sub> O <sub>3</sub>	20	1.0	500	25	3.0	3.8	
12.	Al <sub>2</sub> O <sub>3</sub>	50	1.0	500	25	3.4	9.5	
13.	Al <sub>2</sub> O <sub>3</sub>	80	1.0	500	25	13.8	15.2	

(a) Based on nominal silver content

For Runs 11, 12 and 13 in which the coprecipitant was  $\text{Al}_2\text{O}_3$ , the concentration of the carbon dioxide in the effluent gas as a function of time is shown in Figure 7. It will be noted that the effluent gas in both the 20% and 50% silver oxide preparations contains carbon dioxide immediately, whereas with the preparation containing 80% silver oxide, no carbon dioxide appears in the effluent for 980 minutes.

Further details on the  $\text{Ag}_2\text{O}-\text{Al}_2\text{O}_3$  coprecipitates appear in Table 8. The  $\text{Al}_2\text{O}_3$  content shown in column 4 is calculated from a knowledge of the actual  $\text{Ag}_2\text{O}$  content and the ratio of  $\text{Al}_2\text{O}_3$  to  $\text{Ag}_2\text{O}$  in the preparation. The other material in the preparations as shown in column 5 is probably water. It should be noted that these results are obtained with dried samples and that the amount of water retained in the sample increases as the  $\text{Al}_2\text{O}_3$  content of the sample increases, as might be expected.

For the sample containing a nominal 80% of  $\text{Ag}_2\text{O}$ , breakthrough did not occur for 980 minutes at which point the bed contained 9.6 grams of  $\text{CO}_2$  per 100 grams of bed. In addition it is seen that at saturation, the preparation containing 80%  $\text{Ag}_2\text{O}$  has a ratio of  $\text{CO}_2$  to  $\text{Ag}$  of 0.49, very close to the theoretical value for  $\text{Ag}_2\text{CO}_3$ . This means that all of the silver oxide was available for reaction with the  $\text{CO}_2$ , a most desirable situation. As stated in the introduction, the aim of the work with  $\text{Ag}_2\text{O}$  was to prepare a material having both a high capacity for  $\text{CO}_2$  and a high rate of  $\text{CO}_2$  absorption. The  $\text{Ag}_2\text{O}-\text{Al}_2\text{O}_3$  preparation containing 80%  $\text{Ag}_2\text{O}$  appears to meet these goals.

In the case of the preparation containing 20%  $\text{Ag}_2\text{O}$  nominally, it is seen that the ratio of  $\text{CO}_2$  to  $\text{Ag}$  is greater than 0.5. This may be accounted for by the formation of  $\text{AgHCO}_3$  rather than  $\text{Ag}_2\text{CO}_3$  although this is by no means certain.

The preparations containing 20% and 50%  $\text{Ag}_2\text{O}$  nominally, showed immediate breakthrough. According to Equation 5 in Section III this would seem to mean that the amount of these materials present in the column was less than the critical amount as defined by Equation 5. The preparation containing 50%  $\text{Ag}_2\text{O}$  appears to give an inflection in the breakthrough curve but the reason for this is not yet known.

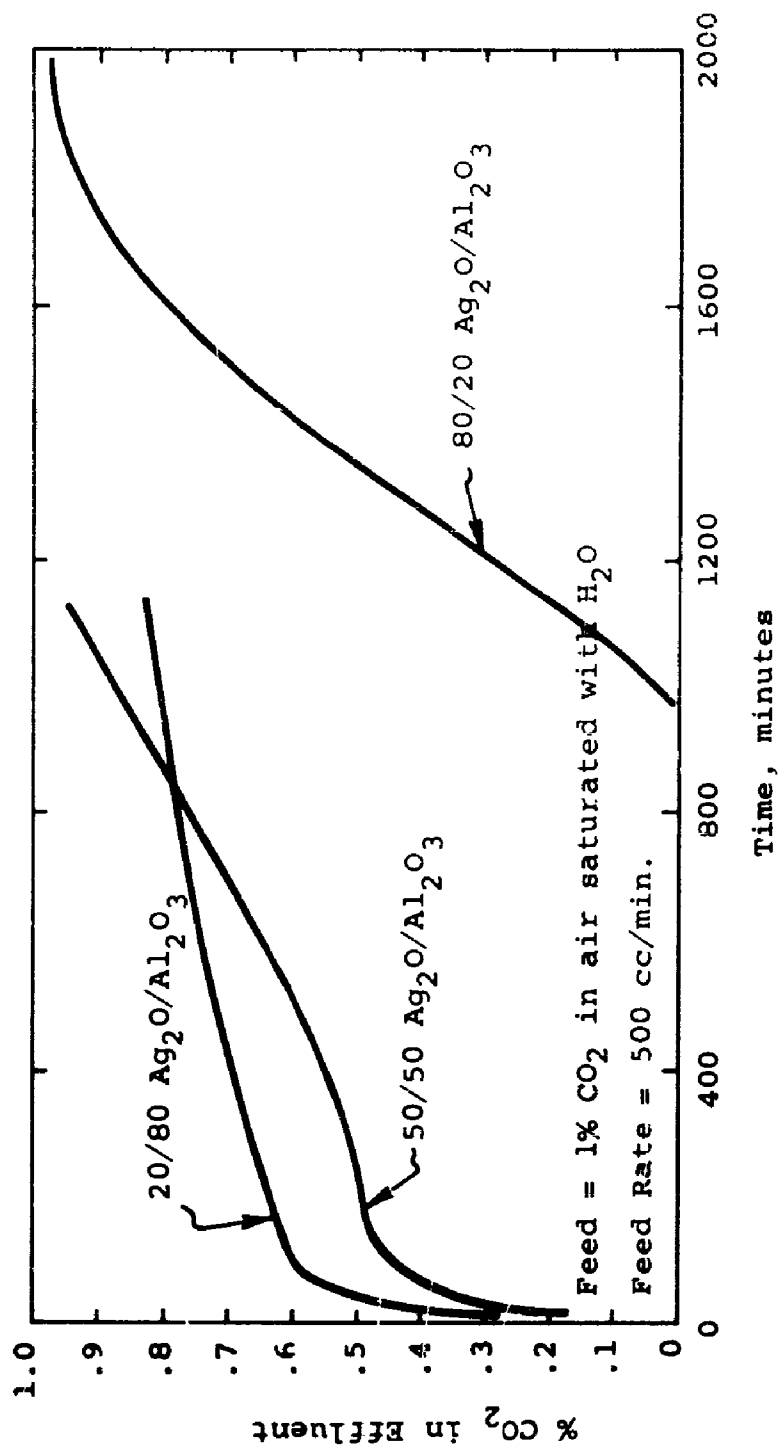


Figure 7. Absorption Curves for  $\text{Ag}_2\text{O}/\text{Al}_2\text{O}_3$  Mixtures



TABLE 8

## ABSORPTION ON

Ag<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> Mixtures

No.	Composition (weight %)				CO <sub>2</sub> Loading			
	Nominal	Actual		Other	At Breakthrough		Total	
	Ag <sub>2</sub> O/Al <sub>2</sub> O <sub>3</sub>	Ag <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub> - <sup>(1)</sup>		%CO <sub>2</sub>	Time (Min.)	%CO <sub>2</sub>	Moles Per g-atom Ag.
11	20/80	7.5	30.0	62.5	0	Immediate	2.97	0.84 <sup>(5)</sup> 1100 <sup>(2)</sup>
12	50/50	27.5	27.5	45.0	0	Immediate	3.38	0.34 1100 <sup>(3)</sup>
13	80/20	73.7	18.4	7.9	9.6	980	13.8	0.49 2100 <sup>(4)</sup>

(1) calculated from Ag<sub>2</sub>O concentrations

(2) ratio of effluent to influent gas concentrations - .83

(3) ratio of effluent to influent gas concentrations - .93

(4) essentially at saturation

(5) CO<sub>2</sub>/Ag - 0.5 max. Theoretical

## VI. CONCLUSIONS

The results of the studies on molecular sieve 5A can be stated as follows:

1. 5A molecular sieve will adsorb  $\text{CO}_2$  from air containing 1%  $\text{CO}_2$  by volume.
2. 5A molecular sieves also adsorb other components present in air such as moisture and, to a small extent, nitrogen.
3. 5A molecular sieves will hold about 3.5% by weight of  $\text{CO}_2$  at the point where  $\text{CO}_2$  is first detected in the effluent from the adsorber when the linear flow rate of air thru the column is about 0.55 ft./sec. This result is higher than that obtained by other workers at comparable flow rates and may be explained by variations from batch to batch of molecular sieve and by differences in the moisture content of the air.
4. The molecular sieve can be almost completely regenerated by sufficient heating and application of vacuum or purging with an indifferent gas.
5. The molecular sieve gives a sharp breakthrough curve (the concentration of the  $\text{CO}_2$  in the effluent increases rapidly once  $\text{CO}_2$  starts to come through the column.)

Since a more detailed study of the operating characteristics of 5A molecular sieve was being made by another group (See Reference 5) the largest part of the effort in this program was devoted to finding ways of utilizing silver oxide successfully. As will be noted from the results and discussion in the previous section, none of the silver oxide preparations except the material prepared by coprecipitation of  $\text{Ag}_2\text{CO}_3$  and  $\text{Al}(\text{OH})_3$  with subsequent decomposition of the  $\text{Ag}_2\text{CO}_3$  to  $\text{Ag}_2\text{O}$  by heating gave both high  $\text{CO}_2$  capacity and good rates of absorption. It should also be noted that these results were obtained using a gas stream saturated with water at room temperature.

We will, therefore, restrict the discussion to these latter preparations. Two things are immediately apparent. In order to obtain a high capacity for  $\text{CO}_2$  absorption, the  $\text{Ag}_2\text{O}$  must be present as a major constituent in the preparation and it must be present in such a form that all of the silver oxide is available for reaction. In addition, it appears necessary to have water vapor present in the gas stream or moisture present in the absorbing bed. The  $\text{Ag}_2\text{CO}_3\text{-Al}_2\text{O}_3$  preparation appears to be the most satisfactory preparation. The  $\text{Al}(\text{OH})_3$  is precipi-

tated as a gelatinous material with the  $\text{Ag}_2\text{CO}_3$  very finely dispersed throughout this gel. Upon drying, the gel develops a high porosity, although it undoubtedly does not lose all of its water. This high porosity plus the fine state of dispersion of the  $\text{Ag}_2\text{CO}_3$  in the preparation are probably responsible for the high activity of this preparation.

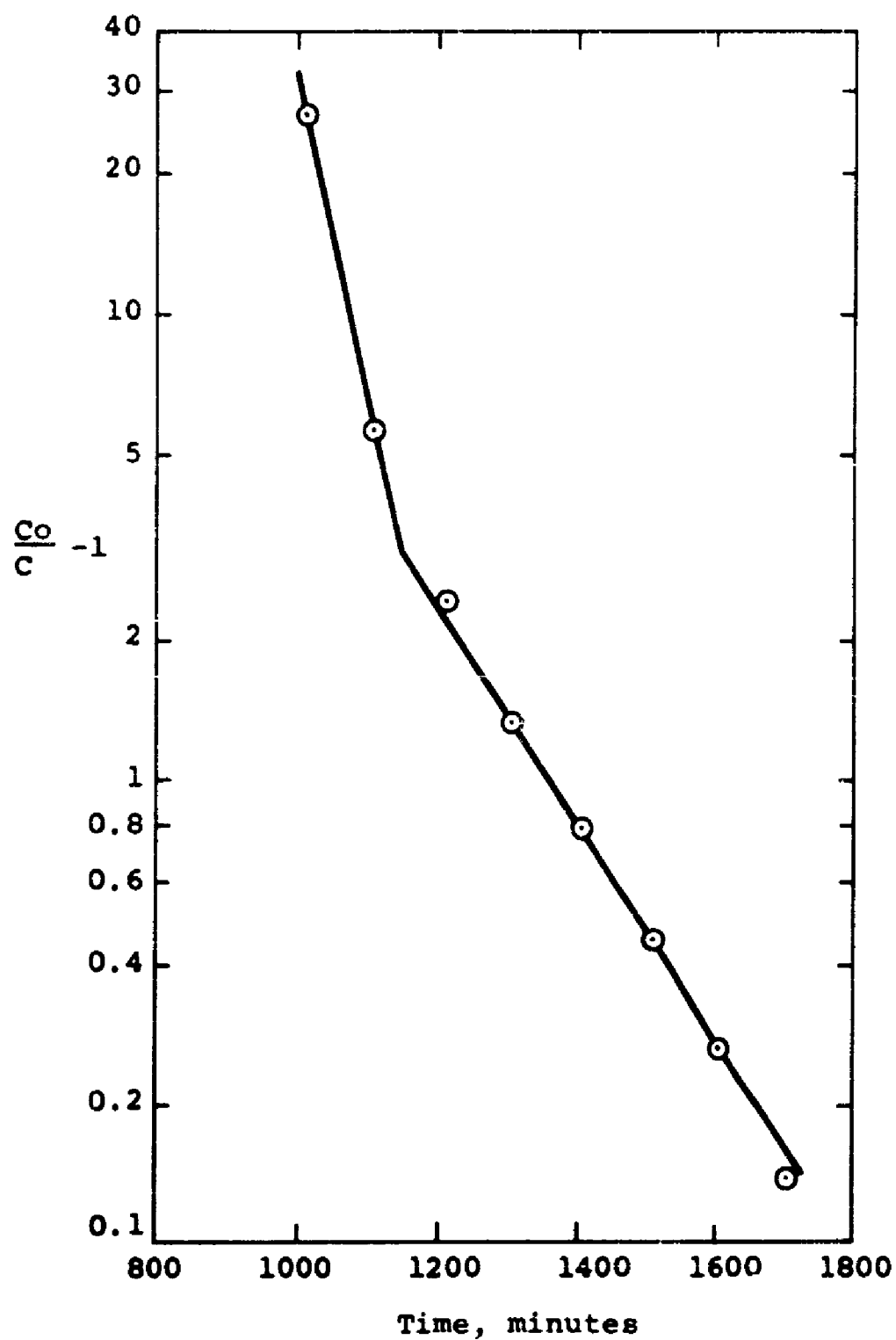
The fact that the  $\text{Ag}_2\text{O}$  is prepared by decomposition of  $\text{Ag}_2\text{CO}_3$  rather than by direct precipitation as the oxide is probably also of importance. The decomposition may serve to develop the porosity of the preparation and may also serve to condition the  $\text{Ag}_2\text{O}$  to reabsorption of  $\text{CO}_2$ . The presence of moisture in the air and the fact that the aluminum hydroxide itself retains a considerable amount of water cannot be overlooked. It is possible that other gels that absorb water and are highly porous might function in the same way as the  $\text{Al}_2\text{O}_3$ .

At saturation, the amount of  $\text{CO}_2$  absorbed by the  $\text{Ag}_2\text{O}-\text{Al}_2\text{O}_3$  preparation containing 80%  $\text{Ag}_2\text{O}$  corresponded almost exactly to the stoichiometric amount of  $\text{CO}_2$  in  $\text{Ag}_2\text{CO}_3$  indicating that all of the silver oxide sites are available for reaction with the  $\text{CO}_2$ .

According to equation 3, Section III, a plot of  $\ln\left(\frac{C_0}{C} - 1\right)$  versus  $T$  should be a straight line. A plot of the data from the run with 80%  $\text{Ag}_2\text{O}$  in Figure 8 shows there are two distinct lines, indicating the presence of two distinct mechanisms at different periods in the run. During the early part of the run it appears that the rate is high and is probably governed by the rate of the surface reaction. After the surface is reacted, the reaction rate decreases and is now limited by the rate at which  $\text{CO}_2$  diffuses through the surface oxide layer.

In Figure 9, the data from this run are plotted as the percent of saturation versus the square root of the time. The data from the middle section of the run fall on a straight line which would be typical of a diffusion controlled reaction. Deviations from the straight line occur at both ends of this time range. The deviation at the beginning is probably due to the fact that the reaction is not diffusion controlled during the early portion of the run. Deviations at the end are probably caused by difficulties in measuring small concentration changes in the column effluent at the end of the run.

Because of the change in reaction mechanism over the course of the run it would be difficult to use the analytical approach for the design of a column. The MTZ approach appears to be more suitable for this material.



**Figure 8. Plot of Data from 80%  $\text{Ag}_2\text{O}$  Preparation According to Equation 3, Section III**

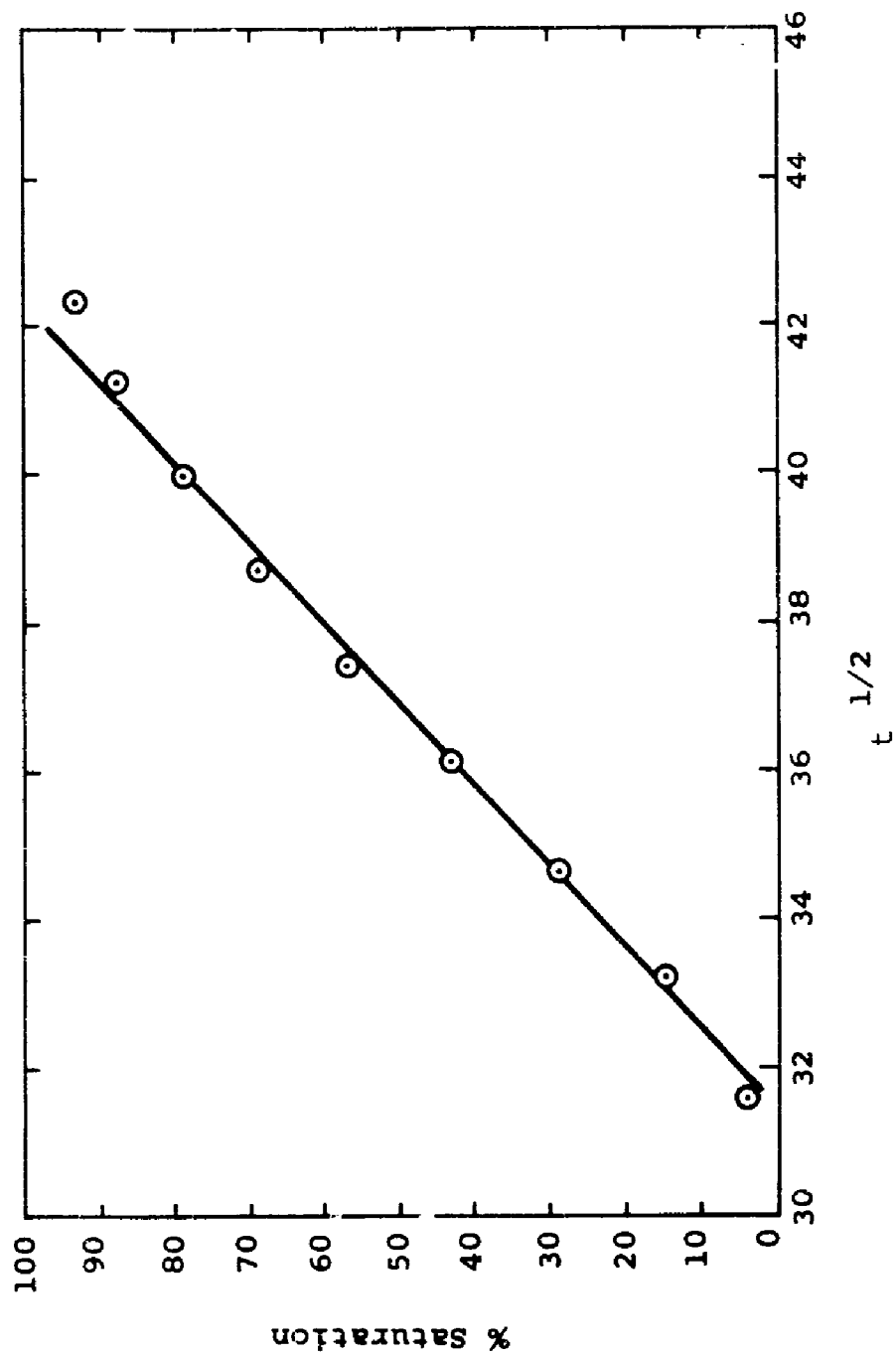


Figure 9. Plot of Data from 80%  $\text{Ag}_2\text{O}$  Preparation

The value of  $Z_a$ , the Mass Transfer Zone can be obtained from the curve in Figure 5 for the 80%  $\text{Ag}_2\text{O}$  preparation. From this curve we obtain:

$$\begin{aligned}\theta_A &= 2000 - 950 = 1050 \text{ minutes} \\ \theta_E &= 2000 \text{ minutes} \\ f &= 0.4 \\ Z &= 8.5 \text{ inches} \\ Z_A &= 6.5 \text{ inches}\end{aligned}$$

This value for the Mass Transfer Zone compares quite favorably with values obtained by others for 5A molecular sieve (See Reference 5).

A further consideration in the use of a regenerable  $\text{CO}_2$  absorber is the ease of regeneration and the conditions under which regeneration can be accomplished. The use of  $\text{Ag}_2\text{O}$  as a regenerable  $\text{CO}_2$  absorber is complicated by the ease with which  $\text{Ag}_2\text{O}$  decomposes to silver. The available data for the decomposition of  $\text{Ag}_2\text{CO}_3$  to  $\text{Ag}_2\text{O}$  and  $\text{CO}_2$  and the decomposition of  $\text{Ag}_2\text{O}$  to  $\text{Ag}$  and  $\text{O}_2$  are shown in Figure 10. It is seen that at any temperature the equilibrium pressure of  $\text{O}_2$  over  $\text{Ag}_2\text{O}$  is higher than the equilibrium pressure of  $\text{CO}_2$  over  $\text{Ag}_2\text{CO}_3$ . It would therefore appear that the  $\text{Ag}_2\text{O}$  would decompose more readily than the  $\text{Ag}_2\text{CO}_3$ . Lewis (ref. 7), in his classic study of the decomposition of  $\text{Ag}_2\text{O}$  has shown, however, that the rate of decomposition is not rapid so that the kinetics of the two decomposition reactions may be the deciding factor rather than the respective equilibria. Preliminary experiments at Isomet have indicated a slow decrease in  $\text{CO}_2$  absorbing capacity over a period of several cycles. The exact reasons for this decline are not yet known.

The results of this work have shown that a silver oxide preparation having a high capacity for  $\text{CO}_2$  and a rapid rate of absorption, can be made. The data indicate this to be the best available absorber in this range of  $\text{CO}_2$  concentrations. Optimum desorption conditions must still be determined.

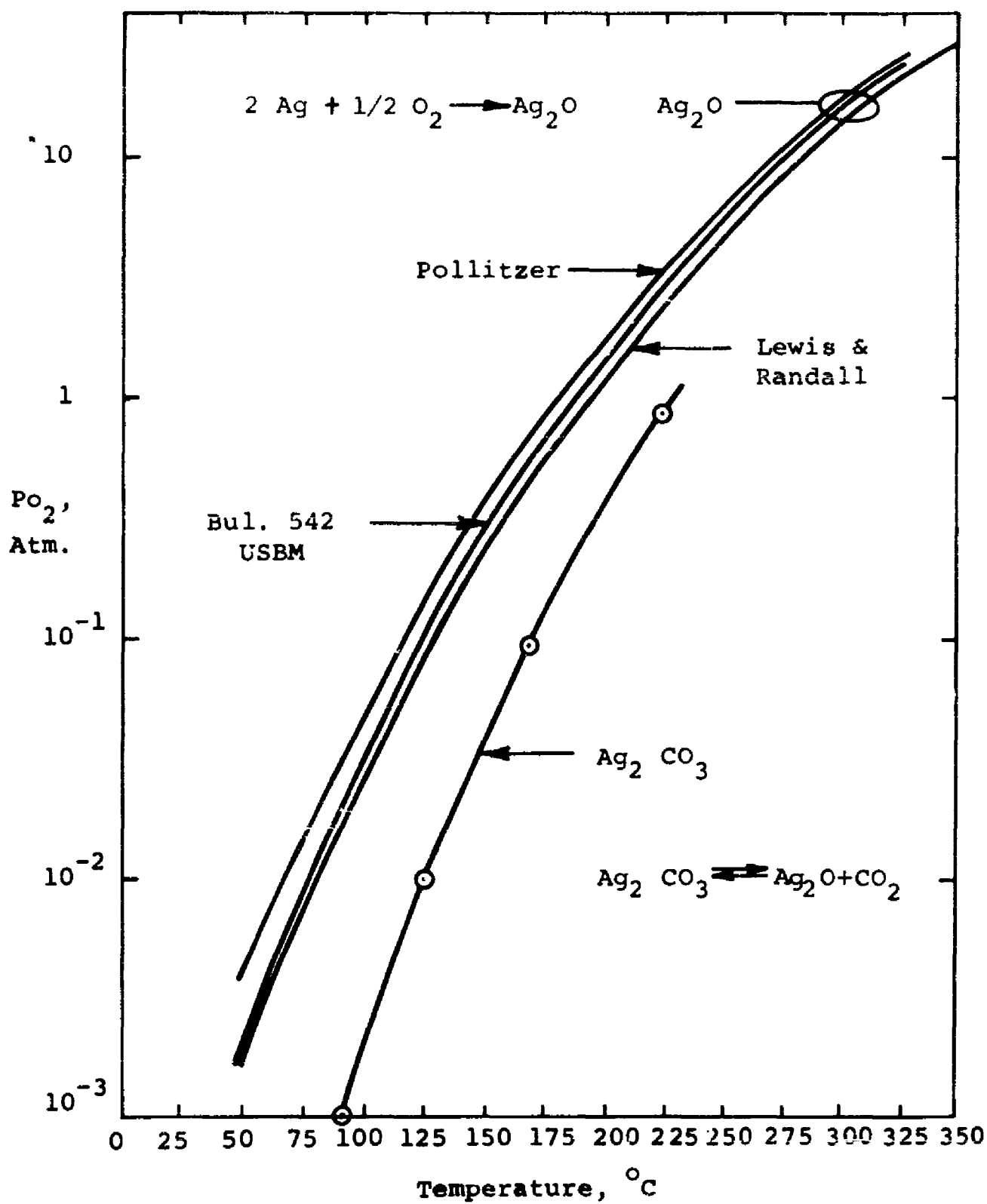


Figure 10. Decomposition Pressures of  $\text{Ag}_2\text{CO}_3$  and  $\text{Ag}_2\text{O}$ .

## VII. Design Considerations

Based on the information available for the 80%  $\text{Ag}_2\text{O}$  preparation an estimate of the size of system required to handle three men can be made. We assume that the system must remove 2.2 pounds of  $\text{CO}_2$  per man per day from an air stream containing 1%  $\text{CO}_2$  by volume at room temperature and 1 atmosphere pressure. This is equivalent to 1.135 liters of  $\text{CO}_2$  per minute for three men. Since the air contains 1%  $\text{CO}_2$  by volume, then 113.5 liters of air must be circulated through the absorber per minute (4.02 cubic feet per minute). By a simple scale-up from our laboratory data obtained with a 1-inch diameter column at 500 cc./min., we find that a column 15 inches in diameter and 8.5 inches long would be required and that it would operate for 950 minutes before breakthrough occurs. This column would contain 50 pounds of the 80%  $\text{Ag}_2\text{O}$  preparation and two of them would be required so that one is regenerated while the other is absorbing. Regeneration within 16 hours would be required.

If operation on a shorter time cycle is possible, then the weight of bed required would be smaller. As an example, if regeneration can be accomplished in 4 hours, then it would be possible to decrease the bed weight to 40 pounds instead of 50 pounds. In no case, at this particular flow rate, could less than about 35 pounds of the 80%  $\text{Ag}_2\text{O}$  preparation be used.

It is, of course, desirable to minimize the weight and volume of bed required. A further study of the effect of flow rate on the length of the Mass Transfer Zone would allow the determination of the optimum flow conditions for minimum weight and volume. This would allow trade-offs between column diameter and column length.

It should also be kept in mind that the air entering the column is kept saturated with water. It has been found that the air emerging from the column is dry and that breakthrough of the water occurs at roughly the same time that breakthrough of the  $\text{CO}_2$  does. It is therefore possible that the single column can serve the dual function of removing  $\text{CO}_2$  from and dehumidifying the air.



### VIII. Recommendations for Further Study

The experimental work described in this report has shown the feasibility of preparing silver oxide in such a fashion that it has a high capacity for  $\text{CO}_2$  absorption and will absorb  $\text{CO}_2$  from an air stream containing 1 percent  $\text{CO}_2$  by volume. Many problems remain to be studied before the utility and limitations of this system for  $\text{CO}_2$  control in closed space cabins can be completely specified. Recommended for further study are the following areas:

1. The rates of decomposition of the  $\text{Ag}_2\text{CO}_3$  and  $\text{Ag}_2\text{O}$  in the  $\text{Al}_2\text{O}_3$  matrix as a function of temperature and pressure. These rates will determine the best operating conditions for regeneration of the  $\text{Ag}_2\text{CO}_3$ .

2. The effect of moisture in the air and in the  $\text{Ag}_2\text{O}$ - $\text{Al}_2\text{O}_3$  preparation on the rate of  $\text{CO}_2$  absorption. To date, all the absorption runs have been made with air saturated with water at room temperature. It is important to know how much humidity is required for efficient operation and what the effect of variations in humidity on the absorption will be.

3. A detailed investigation of the chemical and physical characteristics of the  $\text{Ag}_2\text{CO}_3$ - $\text{Al}_2\text{O}_3$  preparation should be made. Preliminary x-ray diffraction examination of some of the preparations already made indicate the presence of unidentifiable phases. The preparations undoubtedly retain considerable moisture at the temperature and pressures used for drying and it appears that this moisture plays some role in the  $\text{CO}_2$  absorption process.

4. Effect of flow rate on the length of the mass transfer zone. All of the runs to date have been made at the low flow rate of 500 cc. of air per minute, corresponding to a linear velocity of 0.154 feet per second in the 1-inch diameter column. Both higher and lower flow rates should be examined so that a column of minimum weight and volume can be designed.

5. Effect of  $\text{CO}_2$  concentration in the gas stream. All of the runs to date have been made with air at 1 atmosphere and room temperature containing 1%  $\text{CO}_2$  by volume. The effect of variation in both the  $\text{CO}_2$  partial pressure and the total air pressure should be studied since it is possible that both of these factors will vary in the closed space cabins.

6. The effect of particle size on the rate of absorption may be important. Experiments should be run with carefully sized fractions of the  $\text{Ag}_2\text{O}$ - $\text{Al}_2\text{O}_3$  preparations.

7. The chemical and physical stability of the  $\text{Ag}_2\text{O}$ - $\text{Al}_2\text{O}_3$  preparations over an extended number of cycles should be investigated since this will determine the long term utility of the material.

8. The best methods for regenerating the  $\text{Ag}_2\text{O}$  after  $\text{CO}_2$  absorption should be investigated. Included in this should be a study of vacuum, inert gas purging, steam regeneration, heating and combinations of these. The effect of these various techniques on the stability of the preparation should be studied.

9. The effect of other trace gases that may be present in the air on the capacity and stability of the bed should be studied as these may accumulate in the bed and eventually poison it if they are not removed during regeneration.

10. The effect of shock, vibration, high-G loads and weightlessness on this system should be investigated to determine the resistance of the absorber to fracture and abrasion.

11. Finally, after all these other variables have been investigated, this system should be used in closed chamber experiments so as to study its operating characteristics under typical conditions that might be encountered in closed space cabins.

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